



## Production and Characterization of Biodiesel from Mango Seed Kernel collected within Maiduguri Area via Trans-Esterification Process

Musa A. Gasma, Misali J. Madu, and Ephraim T. Bello  
Department of Mechanical Engineering,  
Faculty of Engineering,  
University of Maiduguri, Borno State, Nigeria.

### ABSTRACT

*Irregular supply of fossil fuel, unstable petroleum prices and economy, incomplete combustion of fossil fuel, health risk and environmental hazards are common problems associated with the exploration, transportation and use of fossil fuel. In order to minimize these health and environmental hazards associated with the use of fossil fuel, biodiesel which is biodegradable and non-toxic is produced from mango seed oil through the process of acid esterification and alkali transesterification. Mango seed oil was extracted from mango kernel collected within Maiduguri metropolis and characterized before and after transesterification to determine its suitability for use in biodiesel production. Results obtained from the transesterification of 630 ml of oil extracted from 8.5 kg of dry mango powder at 60 °C revealed that the biodiesel obtained from mango seed oil has an acid value of 0.96 mgKOH/g, oil yield of 14.89 %, flash point of 155°C, pour point of 6.17 °C and viscosity of 5.3mm<sup>2</sup>/s at 60°C. Therefore, considering the results obtained, the mango seed oil can be a potential feedstock for biodiesel production.*

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### INTRODUCTION

Biodiesel is an alternative fuel made from renewable biological sources such as vegetable oils and animal fats. According to the US standard specification for biodiesel (American Society for Testing and materials (ASTM D6751), biodiesel is defined as a fuel comprised of mono alkyl esters of long chain fatty acids from vegetable oils or animal fats. The dominant bio-diesel production process, namely transesterification (Romano *et al.*, 2011), typically involves the reaction of an alkyl-alcohol with a long chain ester linkage in the presence of a catalyst to yield mono-alkyl esters (bio-diesel) and glycerol.

Biodiesel presents several advantages such as non-toxicity and eco-friendly properties (Zahan, 2018). The quality and efficiency of biodiesel is found to be more significant than petro-diesel (Saxena *et al.*, 2013). Numerous studies on biodiesel from different sources have been reported.

Nathaniel (2013), extracted pork oil through direct rendering; the research was carried out on the physicochemical properties and characterization of the pork oil. The study revealed that the physicochemical properties such as the refractive index, saponification value, percentage free fatty acid content, iodine value, peroxide value, melting point, acid



value, moisture contents, and hydroxyl values were observed by Gas Chromatography and Mass Spectrometry (GC-MS) to be favorable and within the recommended edible oil fatty acid compositions which are: Oleic acid – 46%, Palmitic acid – 28.19%, and Stearic acid 19.45%.

Kaisan (2017), obtained biodiesel from wild grape (*Ianneamicrocarpa*) seed oil using soxhlet extraction, N-hexane was utilized in the soxhlet extraction method with oil yield of 33.2% and 2.58% of low free fatty acid recorded. Additionally, extraction via mechanical press extraction methods revealed 12.5% oil yield and 42% free fatty acid respectively. Esterification of the extracted wild grape seed oil, revealed free fatty acid reduction from 42% to 2.52% with an oil yield increase of 37.5% while other properties like density remained unchanged. It was observed that mechanical method offers better production process in reference to; production time, oil yield and industrial applications. Zahan (2018), stated that various vegetable oils such as the edible and non-edible types have been used for biodiesel production. These are: palm oil, soya beans oil, corn oil, coconut oil, canola oil, sunflower oil, castor oil and fish oil (Ong *et al.*, 2013). Currently, the quantity of biodiesel produced from these oil crops is not capable of providing biodiesel for commercial purposes. This paper presents the production of biodiesel derived from mango kernel as an alternative energy source in producing biodiesel through transesterification process.

## MATERIALS AND METHOD

### MATERIALS

The major material used for production of biodiesel in this study is

mango seeds and other chemicals and equipment used include:

**Chemicals and Reagents:** The following reagents were used in this study: n-hexane: 99% purity, Methanol: 99.55% purity, Potassium hydroxide: 55% purity, Silica-Gel, Phenolphthalein, Hydrochloric Acid: 35-38% purity, Sodium Sulphate and Sodium Hydroxide.

**Instruments and Apparatus:** The equipment and apparatus used in this study include: laboratory oven, weighing balance, measuring cylinders, stop watch, separating funnel, thermometer, magnetic stirrer and spatula.

### METHOD

#### Sample Collections and Preparation

Mango fruit seeds were collected from waste trash within Maiduguri Metropolis Borno State, Nigeria. The mango kernel seeds were sun dried for a week prior to manual removal of the seeds using sharp knife from pulps so as to reduce the moisture content. It was then dried under roof shade for additional one week until it was completely dehumidified at 33°C-38°C. The mango seeds were sorted to remove unwanted kernel seeds. The dried seeds were crushed into smaller sizes using mortar and pestle. After which it was grounded into powder and sieved to obtain fine powder.

#### Oil Extraction

Oil was extracted from Mango seed using Soxhlet extractor. A mixture of extracted mango oil and N-hexane solvent was collected and separated by simple distillation. During the oil extraction process a total of 8.5kg of mango powder was used and a total of 630ml of mango oil was extracted as shown in Plate1.

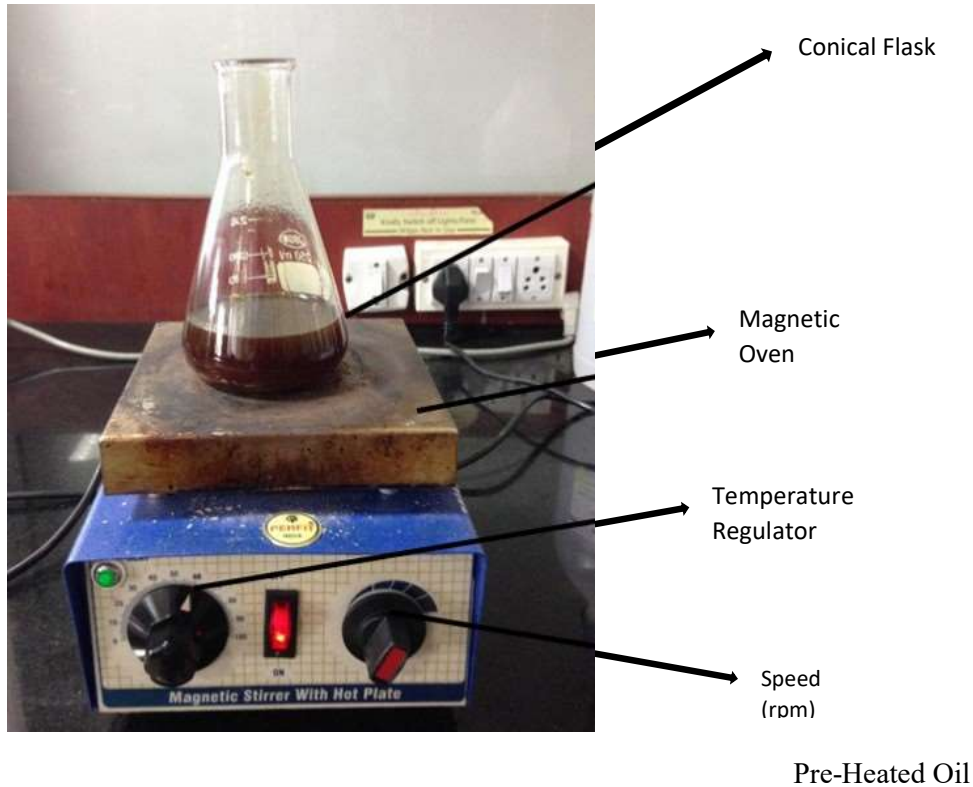


Plate 1: Extracted mango oil on a hot plate

**ANALYSIS AND CHARACTERIZATION OF EXTRACTED MANGO KERNEL OIL**

**Percentage Yield Determination**

The weight of the oil produced and the residue were measured to ascertain the percentage of the oil content. The oil extracted was weighed and the percentage oil yield was calculated by using Equation 1 (Ong *et al.*, 2013).

$$\frac{\text{Oil yield}}{\text{Weight of sample}} = \frac{\text{weight of oil}}{10}$$

**Acid Value Determination**

5% of ethanol was boiled in a water bath to remove dissolved gasses. The

solution was then neutralized by adding few drops of 1% phenolphthalein indicator and 0.1m KOH until a pale pink color was obtained. 10 grams of the extracted oil was added to 50 cm<sup>3</sup> of the hot alcohol mixture and further heated. The mixture was then titrated against 0.1m KOH until pale pink color was obtained. Equation 2 shows how the acid value was calculated (Ong *et al.*, 2013).

$$\text{Acid value} = \frac{M \times V \times 56.1}{W} \quad 2$$

Where, M = Molarity (mol/dm<sup>3</sup>)

V= titer value (cm<sup>3</sup>)

W= weight of oil in

(grams)



### Saponification Value Determination

3 grams of extracted oil sample was weighed in a conical flask fitted with a reflux condenser. 0.1m<sup>3</sup> ethanoic KOH solution was added and the mixture was heated for 30 minutes until the oil dissolved in the solution. The hot soap solution was slowly titrated with 0.5 m HCL using phenolphthalein as an indicator to determine the excess of KOH. The saponification values are were computed using equation 3 (Ong *et al.*, 2013).

$$\text{Saponification value (s)} = \frac{56.1 \times M (V_2 - V_1)}{W} \quad 3$$

Where, W = Weight of oil sample (grams)

V<sub>1</sub> = Vol. of oil sample (cm<sup>3</sup>)

M = molarity of base used (mol/dm<sup>3</sup>)

56.1 = Molar mass of KOH

V<sub>2</sub> = volume of HCL used in the test (cm<sup>3</sup>)

### Determination of Density

To determine its density, equation 4 was used (Jibrilet. *al.*, 2012; Ong *et al.*, 2013)

$$\text{Density (p)} = \frac{W_2 - W_1}{V} \quad 4$$

Where W<sub>2</sub> = Weight of bottle and oil (in gram)

W<sub>1</sub> = Weight of graduated cylinder (in gram)

V = Volume of oil (cm<sup>3</sup>)

### Determination of Viscosity

The viscosity was determined with an Ostwald viscometer that measured the time for a fixed volume of the oil/biodiesel to flow under gravity through a capillary at a temperature of 30 °C (Bello *et al.*, 2011).

### Preparations of Reagents/Solutions

**Table 1:** shows how major reactants were produced in the laboratory.

S/N	Reagents	Method of Preparation
1	0.1mol of Sodium hydroxide (NaOH)	0.35gof NaOH was dissolved in 50cm <sup>3</sup> of distilled water and the solution transferred into a 1dm <sup>3</sup> volumetric flask
2	5% of Methanol Solution	5ml of ethanol was initially dissolved in 100 cm <sup>3</sup> of distilled water
3	0.1mol/dm <sup>3</sup> of methanolNaOH solution	2.8 grams of NAOH was dissolved in 100 cm <sup>3</sup> of ethanol and the solution further mixed with methanol solution
4	1 % Phenolphthalein indicator solution	1gram of phenolphthalein was dissolved in 5 mol/dm <sup>3</sup> methanol and the mixture was dissolved in 100 cm <sup>3</sup> of distilled water
5	0.5mol/dm <sup>3</sup> of Sulphuric Acid	4.3 cm <sup>3</sup> of concentrated HCL was dissolved in 100cm <sup>3</sup> of distilled water and the mixture kept in a fume cupboard

### BIODIESEL PRODUCTION

In this study, catalysts were utilized to increase the speed of the reaction and also to improve the quality of the produced biodiesel. The amount or types of catalysts used depend on the amount of free fatty acid present in the extracted oil (the presence of free fatty acid leads to the formation of soap which

decreases the efficiency of biodiesel). From the study, the amount of free fatty acid present in the extracted mango oil was approximately 10.7%. The biodiesel production via trans-esterification process was carried out in two stages:

- A. The first stage includes an acid catalyzed esterification reaction



- B. The second stage include base catalyzed trans-esterification reaction

**Mango Oil Biodiesel Transesterification Process (two stage process)**

**First stage (Acid Catalyzed Transesterification Reaction)**

- The 500 ml of extracted mango oil was placed in a conical flask and heated to 60 °C.
- Acid catalyzed mixture of 95 ml of Methanol and 5 ml Sulphuric acid was introduced to the heated oil.
- The mixture was stirred for further 35 minutes using a magnetic stirrer.
- After stirring, the mixture was cooled for 5-6 hours for impurities to sediment and then collected using a separating funnel.

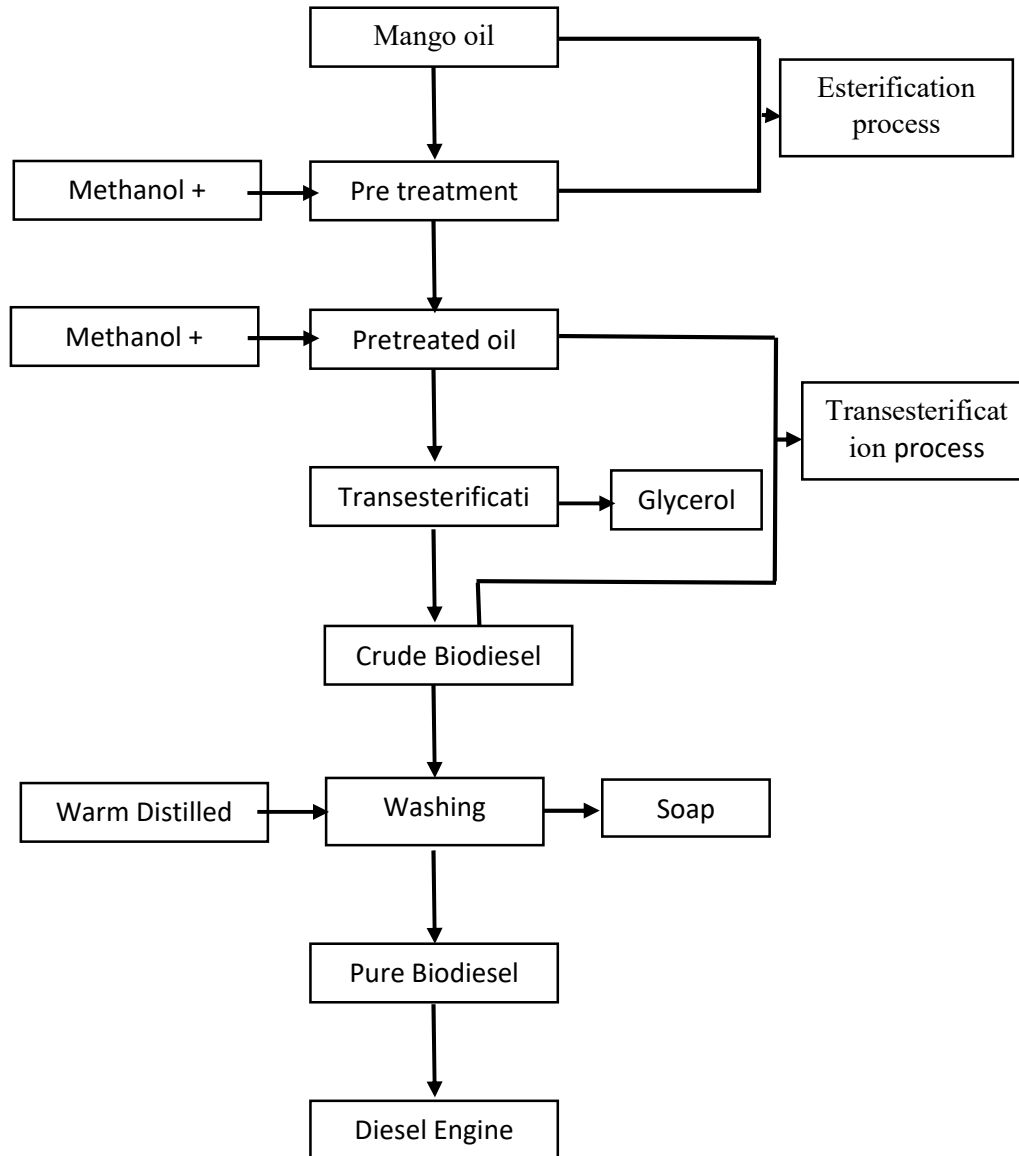
**Second stage (Base Catalyzed Transesterification Reaction)**

- A 400 ml of cooled oil was pre-heated to 65 °C and a base catalyzed mixture of 135.2 ml of Methanol and 3.5g of Sodium

hydroxide NaOH was added to the heated oil.

- The heated oil was heated and stirred for an hour at 60 °C using a hot plate.
- The mixture was cooled for 24 hours forming two distinct layers: the upper layer is 70 % methyl ester known as biodiesel and the lower layer is the glycerol.
- The lower layer (glycerol) was drained using a separating funnel leaving the methyl ester (biodiesel) layer.
- The newly formed biodiesel was further purified using distilled water at 50 °C to remove unreacted methanol and sodium hydroxide.

Additional cleaning procedures like: addition of 13 grams of Sodium Sulfate ( $\text{Na}_2\text{SO}_4$ ) pellets to absorb water and further heating of biodiesel at 70 °C for 30 minutes were performed. Figure 1 illustrates the biodiesel conversion pathway from mango kernel seed.



**Fig 1.** Flow chart of Biodiesel production from Mango Kernel Oil

## RESULTS AND DISCUSSION

### Results

The oil contents of agricultural products provide insight on the susceptibility of oil extraction for industrial processes. The extracted oil yield from

mango seed is 14.89 %. This is lower than the values for soybeans and cottonseed (19-21% and 15-20% respectively) (Nzikou et al., 2010). Also, it is lower than jatropha seed (28-32%), castor seed (55%), rubber seed (68.0%), coconut (60.0%), and castor seed



(67.7%) from studies conducted by Shukla *et al.*,(1992); Pramanik, (2003);Conceição *et al.*,(2007) respectively. According to report by Akinoso *et al.*, (2010) the Food and Agriculture Organization statistics (FAO)

revealed that oil seed should have oil yield higher than 17% to be considered oil feedstock. Result from the characterization raw mango seed oil after its extraction is presented in Tables 2-5.

**Table 2:** Percentage of free fatty acid (FFA) of Mango Seed oil before the Process of Esterification

Properties	Values	Unit
<b>Acid value</b>	38.70	mgKOH/g
<b>Free fatty acid (FFA)</b>	10.7	mgKOH/g

**Table 3:** Property of Mango Seed Oil

Properties	Values	Unit
<b>Specific gravity (S.G)</b>	0.87	Kg/m <sup>3</sup>
<b>Saponification value</b>	77.1	mgKOH/g
<b>Ph</b>	4.50	
<b>Viscosity</b>	6.4	mm <sup>2</sup> /s
<b>Density</b>	0.9	g/cm <sup>3</sup>

**Table 4:** Percentage of Free Fatty Acid (FFA) of the Mango Seed Oil after Esterification

Properties	First Esterification value (mgKOH/g)	Second Esterification value (mgKOH/g)	Third esterification value (mgKOH/g)
<b>Acid value</b>	8.13	5.05	1.094
<b>Free fatty acid</b>	4.07	2.525	0.547

**Table 5:** Comparison between Mango biodiesel, ASTM D6751 Standards and European Standard (EN 14214)

Properties	Biofuel values derived from Mango kernel	ASTM & EN STANDARDS (Zahan, <i>et al.</i> ,2018).		
		ASTM for Biodiesel	ASTM for Fossil Fuel	European Standard for Biodiesel
<b>Yield (%)wt</b>	74.0±4.56	96	-	-
<b>Viscosity (mm<sup>2</sup>/s) at 40°C</b>	5.3±0.35	1.9-60	1.3-4.1	3.5-50
<b>Density (g/cm<sup>3</sup>) at 20°C</b>	0.88	0.79-0.91	0.82	0.86-0.90
<b>Acid value (mgKOH/g)</b>	0.96	0.8	0.21	-
<b>Cloud point (°C)</b>	-7.67±0.74	-3 to -12	-15 to -5	-
<b>Flash point (°C)</b>	155	100-170	65	≥120
<b>Pour point (°C)</b>	-6.17±0.62	-15 to -16	-35 to -15	-



## DISCUSSION

### **Free fatty acid (FFA)**

Fatty acid value of 10.7 mg KOH/g was recorded (Table 2), this value is quite high and suggests further pretreatment (esterification) procedures are required to reduce free fatty acid (FFA) content before base catalyzed transesterification. Leung *et al.*, (2010) revealed that high free fatty acid content will result in low biodiesel yield. The extracted mango oil was subjected to the acid catalyst esterification up to three times and obtained the value of 4.07mg KOH/g, 2.525mg KOH/g and 0.547mg KOH/g respectively as shown in Table 4, before the base catalyst transesterification

### **Kinematic Viscosity**

Kinematic viscosity is a measure of resistance of fluid to flow under the influence of gravity. The result from this study shows that viscosity of biodiesel produced from mango seed oil was 5.3(mm<sup>2</sup>/s) as shown in Table 5 juxtapose to the ASTM D6751 standard of 1.9 - 60 mm<sup>2</sup>/s it falls within the ASTM norms.

Viscosity of fuel relates to the fuel lubricity. Low viscosity fuels are unlikely to provide satisfactory lubrication in fuel injection pump. High viscosity in fuel is responsible for atomization of fuel, incomplete combustion, choking of injector thereby forming larger droplets on injector, ring carbonization and accumulation of the fuel in the engine. The result from this study indicates the susceptibility of mango feedstock for biodiesel production (Umaru *et al.*, 2014).

### **Density**

From the oil and biodiesel characterization is shown in table 5. It was observed that the average densities of extracted oil and biodiesel samples (0.9 and 0.88 g/cm<sup>3</sup> respectively) fall within the range specified by ASTM D6751 (0.79- 0.91 g/cm<sup>3</sup>). It could be seen that the mango

biodiesel value is closer to the higher limit (0.91 g/cm<sup>3</sup>); this is due to the fact that the biodiesel is made out of plant oil.

### **The Acid Value**

The acid value of mango biodiesel (0.96mgKOH/g) is tabulated in Table 5. This value is slightly higher than ASTM D6751 standard of 0.8mgKOH/g. Acid value is an indicator of free fatty acid content in methyl ester. It indicates the corrosive nature of the fuel, its filters clogging tendency and the amount of water that may be likely present in the biodiesel. This parameter also measures the freshness of the biodiesel. The higher the acid value the lower the quality of the fuel. (Umaru *et al.*, 2014).

### **The Cloud and Pour Point**

The cloud and pour point are criterion used for low temperature performance of a fuel. This work characterized the biodiesel from mango seeds oil and obtained the values 7.67 °C and 6.17 °C for cloud and pour point respectively as shown in Table 5. Both values fall out of range agreement of ASTM D6751 biodiesel standards (-3 to -12 °C and -15 to -16 °C respectively). Cloud and pour values indicate the behavior of the biodiesel under a specified climate condition. Both values obtained from mango biodiesel negate the prospect of mango feedstock for biodiesel production in cold climatic region. (Umaru *et al.*, 2014).

### **Flash Point Temperature**

Flash point is the minimum temperature at which a fuel must be heated for it to ignite air/vapor mixture. The ASTM D6751 standard flash point temperatures of biodiesels range between 100-170 °C as shown in Table 5. However, the U.S. Department of Transportation specified 90 °C as the flash point for non-hazardous fuel. This study revealed the flash point of





mango biodiesel as 155 °C (Table 5). This value is consistent with ASTM D6751 standard however significantly higher for non-hazardous fuel. The mango biodiesel value (155 °C) signifies that the biodiesel is free of methanol as traces of methanol significantly lowers flash point temperature and negatively affects diesel engines parts such as fuel pumps and nozzle seals (Umaru *et al.*, 2014).

### CONCLUSION

In this paper, biodiesel was produced from mango seed oil through the process of transesterification using KOH as catalyst. Oil yield of 14.87% was obtained and properties such as viscosity, density, acid value and flash point temperature with exception of cloud and pour points of biodiesel from mango kernel all fulfilled the European standard (EN 14214) and American Society for Testing and Material (ASTM D6751 standards) of biodiesel properties. Therefore, mango seed oil can be suitable for the production of biodiesel which could serve as a substitute for other sources of fuel.

### RECOMMENDATION

A laboratory analysis on mango biodiesel should be conducted using test engines to evaluate the performance efficiency of the fuel which will lead to the optimization of mango seed biodiesel.

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Corresponding author: Gasma, M. A. ✉ [ephraimbello17@gmail.com](mailto:ephraimbello17@gmail.com) ✉ Department of Mechanical Engineering, Faculty of Engineering, University of Maiduguri. © 2019 Faculty of Tech. Education, ATBU Bauchi. All rights reserved



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